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HUGONIOT MEASUREMENT AND COMPARISON WITH STATIC BEHAVIOR**

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**DYNAMIC COMPACTION OF ALUMINUM NITRIDE POWDER:
HUGONIOT MEASUREMENT AND COMPARISON WITH STATIC BEHAVIOR***

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Abstract

We have measured the shock Hugoniots of two unsintered aluminum nitride powders having initial densities of 1.30 g/cm^3 and 1.53 g/cm^3 . Stresses achieved in our experiments range from 0.25 to 1.8 GPa, corresponding to linear compaction rates of 0.3 to 0.6 km/s. We find that densification is incomplete behind the first shock wave, proceeding only to about 70% of the solid density regardless of the initial density. Upon reshock, however, significantly higher densities are achieved. Initial compaction of the powder to a relative density of 65 to 70% occurs readily at stresses below 0.25 GPa. For greater stresses, however, densification is slight. Comparisons with static compression data on the same powders suggest that this resistance to compaction is an effect of compaction rate.

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I. Introduction

The static compaction behavior of ceramic powders has been the subject of several studies.¹⁻⁶ Some recent work, however, has explored the dynamic, or shock wave, compaction of ceramic powders, both as a technique for the production of fully dense, well-bonded materials⁷⁻¹² and as a means of inducing high-pressure and high-temperature phase changes.¹³⁻¹⁵ The analysis of the dynamic experiments rests in large part on a knowledge of the shock response of the powders under study, for it is from the locus of states achieved upon shock loading, the Hugoniot curve, that the densities and energy densities behind the various shock waves are deduced. The latter quantity is of particular concern, as it determines the type and extent of local microstructural changes in the powder.¹⁶ Hugoniot data for a number of porous ceramics are given in Ref. 17. Some data for silicate minerals are also available in the geophysical literature,¹⁸⁻¹⁹ applied to the interpretation of impact and high-pressure wave-propagation phenomena. Information on technical ceramics, however, is in large part restricted to high stresses, in excess of 10 GPa,^{17,20,21} which are suitable for equation-of-state determinations but may be greater than needed merely to achieve full density or adequate cohesion. Some data at lower pressures are available for MgO and Al₂O₃.^{21,22}

Aluminum nitride undergoes a brittle-to-ductile transition when loaded triaxially,²³ and shows considerable plasticity during dynamic compaction.⁹⁻¹¹ Its dynamic properties are thus of particular interest. In this paper, we present our measurements of the Hugoniots of two aluminum nitride powders having different initial packing densities. We first describe our experiments and starting materials. We then present our results and analyze them to show that complete densification is not achieved upon passage

of the initial shock, and that reflections of the initial shock ("reshock") produce significant increases in the compact density. The variation of density with dynamic stress is presented and discussed in relation to static compression data. We note in particular what appear to be compaction-rate effects.

II. Experimental Procedure

The experiments in this study were conducted with a helium-driven 6.35-cm-bore light gas gun. Aluminum 6061 projectiles of various weights were used, and the projectile speed could be varied up to a maximum of 800 m/s. Projectile speed was determined to within 1 to 2% using a system of electrically shorting whisker pins placed in the muzzle just in front of the sample.

The sample was positioned at the end of the muzzle in the assembly shown schematically in Fig. 1. The powder was packed by means of a vibrator into the 0.2-cm gap between a 0.3-cm-thick cover plate and a 1.27-cm-thick back plate (both of aluminum 6061). The use of the same material for projectile and cover produces a single, well-defined shock in the powder, simplifying the analysis. The high aspect ratio of the arrangement insured that this shock was one-dimensional near the center of the sample. Thin-film carbon piezoresistive shock pressure sensors* were affixed to the cover and back plates (Fig. 1) and were used to record the initial shock stress and the reshock stress in the compact. However, primary emphasis was placed on

* Dynasen, Inc., Goleta, CA. The change in resistance of these sensors with stress is measured with a bridge circuit, of which it forms one leg. The voltage across the bridge is recorded on an oscilloscope, and with suitable calibration this voltage - time record may be converted directly to a stress - time history.

measurements of shock transit time and impedance matching²⁴ in determining the shock states of the powder, because of their freedom from calibration errors. The transit time between the two carbon sensors was measured directly, and the thickness of the specimen was taken to be the distance between the midplanes of the sensors. The relative tilt of the sample and projectile at impact was measured with three piezoelectric sensing pins spaced equally around the rim of the cover. The tilt was typically 2.5 mrad. All experiments were conducted in a vacuum of 100 millitorr or less.

The hydrostats of the powders were obtained using the method discussed in Ref. 25. Samples were sealed in cylindrical tin containers 1.27 cm in diameter by 2.54 cm in length and compressed in a piston-cylinder die, end-loaded in a double-acting press.²⁵ The very low yield strength of the tin produces conditions which are very nearly hydrostatic, and the sample compression at pressure can be deduced from the ram displacement. Corrections for die distortion and friction are obtained from separate experiments on a solid nickel standard. The relatively large size of the static specimens compared with the 0.2-cm-thick samples used in the dynamic measurements produced higher initial packing densities in the static experiments (1.96 g/cm³, 60%, for powder I; 1.79 g/cm³, 55%, for powder II).

Aluminum nitride powders were obtained from two different commercial sources, and the properties of each powder are summarized in Table I. The particles of powder I consist of micrometer-sized agglomerates of ~0.1- μ m crystallites, as shown in the micrograph of Fig. 2. Powder II has a similar character. Neither powder is pure, but the 4-6% contamination (principally oxides or oxy-nitrides) is typical and is expected to have little effect on their dynamic response.

III. Results

A representative sensor record is shown in Fig. 3. The initial stress in the powder, at the front sensor (Fig. 3a), rises within 160 ns to a peak and then relaxes to a plateau. The peak is probably a capacitive effect common to sensors of the type used here,²⁶ but may also be related to the porosity of the specimens. The value of the stress at the plateau was in all cases taken to represent the stress in the compacted powder behind the initial shock front. This stress remains constant until the return of the reflected wave, showing that lateral release does not occur in the center of the specimens in the time span of interest here, and that the initial compaction occurs under average conditions of one-dimensional strain. Conditions behind the initial shock are summarized in Tables II and III.

The reshock stress, reflected at the back of the assembly (Fig. 3b), rises rapidly to a much higher level than the initial shock. The reshock stresses given in Tables IV and V have been corrected for errors in calibration, which have been reported for these sensors at stresses in excess of about 2.0 GPa.²⁷ The records provide no clear evidence for a lower-amplitude stress wave that precedes the main compaction shock ("precursor"), such as observed in experiments with pressed or sintered porous metals.^{28,29} We attribute this to the low density and lack of cohesion of the initially unsintered powder. It should be noted, however, that the resolution of our measurements is limited to 100 to 200 ns by the sensor thickness.

The Hugoniot curves we obtained for the two powders are shown in the stress - material speed plane in Fig. 4. We find satisfactory agreement between the impedance-matching results and the corrected direct sensor measurements in the stress range produced by the initial shock, in accord with previous work.²⁷ Also shown in Fig. 4 are the states achieved upon reshock,

all of which necessarily lie along the Hugoniot of the aluminum 6061 back plate.²⁴ The slopes of the reshock Rayleigh lines are significantly less than that of solid AlN. Reshock data are summarized in Tables IV and V.

No attempt was made to examine material recovered from these experiments. Material recovered from similar experiments, however, showed that the compacts were badly fractured and had little strength. The densities of these specimens, as determined by mercury porosimetry, ranged from 79% to 85% of the solid. These densities are in accord with those derived from Hugoniot data in the following section.

IV. Analysis

The presentation of the Hugoniot in the plane of the stress, P , and the average material speed behind the shock, U_p , is particularly convenient because these data are obtained directly from both the sensor and the impedance-matching measurements. The Rankine-Hugoniot relationships, however, relate P and U_p to the shock speed, v_s , and the specific volume, V , so that given any two of these variables, the other two may be calculated. These relationships are derived and discussed extensively elsewhere²⁴ and are summarized briefly for convenience in the Appendix.

Equation (A-4) may be used directly to determine the specific volume behind the shock, or we can combine Eqs. (A-1) and (A-2) to yield

$$(U_p - U_0)^2 = (P - P_0) (V_0 - V) \quad , \quad (1)$$

where the subscripts denote the initial states in front of the shock. For the initial shock, $U_0 = 0$ and $P_0 = 0$, so that Eq. (1) reduces to:

$$V = V_0 - \frac{U_p^2}{P} \quad . \quad (2)$$

The specific volumes and densities ($1/V$) derived from Eqs. (1) and (2) are collected in Tables II through V, and the relative densities from impedance matching only are plotted as a function of stress in Fig. 5. Within the stress range studied there is relatively little change in the density of the compact behind the initial shock. It should be noted that these are the densities at the stress indicated, and as such differ from the usual values quoted for compacted powders after the stress has returned to ambient. They do not, therefore, include increases in the specific volume which may occur as a result of pore or crack opening during release. The large error bars shown in Fig. 5 result from the dependence of V on the square of the material speed and the inverse of the stress in Eqs. (1) and (2). A relatively modest error in these quantities translates into a substantially larger error in the calculated specific volume.

The densities given in Table II and III show that complete consolidation is not achieved behind the initial shock. This is apparent from the comparison in Fig. 4 of the experimental Hugoniot curves and those derived assuming that complete densification of the powder occurs regardless of stress. Significant increases in density also occur upon reshock, Tables IV and V, although these are not sufficient to fully consolidate the powder.

A least-squares analysis of the data for the 1.53-g/cm^3 powder, which shows the least scatter, yields a linear relationship between the stress and the relative specific volume of the powder:

$$\rho_r = V_{\text{solid}}/V = 0.685 + 0.017P \quad , \quad (3)$$

where P is in GPa. This relationship may be used in a description of the

dynamic compaction of porous materials^{30,31*} to produce the curves indicated in Figs. 4 and 5. There is little statistical significance to Eq. (3), however, and data for both initial densities can be equally well described by a constant value of 0.70.

This behavior contrasts with the static compression curves, also shown in Fig. 5. These are typical of ceramic powders, and show a relatively large initial slope, $d\rho/dP$, which decreases quickly and continuously as the pressure and density increase. Within the range studied, the initial density has little effect on the static high-pressure behavior of the powder.

V. Discussion

The reshock states summarized in Tables IV and V and shown in Fig. 4 are determined by the intersection of the aluminum 6061 Hugoniot and the second, or reshock, Hugoniot of the powder compact, reflected about the material speed of the first shock state.²⁴ Although we cannot reconstruct this reshock Hugoniot in detail from our data, it is obvious from Fig. 4 that the locus of reshocked states is qualitatively similar to the principal Hugoniot of the powder and is considerably different from that of the solid. This observation is consistent with a relative density of ~70% behind the initial shock (Tables II and III), for which we expect the reshock Hugoniot to be comparable to, but slightly steeper than, the principal Hugoniot.

* These models actually describe the powder consolidation in terms of a $P-\alpha$ relationship in which α is the ratio V/V_{solid} , where V_{solid} is the volume of the solid at the stress specified. This is slightly less than the solid specific volume under ambient conditions, but at the low stresses considered here the difference is negligible.

The linear compaction rates of the dynamic experiments correspond to the material speeds behind the primary shock waves, and range from 0.3 to 0.6 km/s. These very high values suggest that rate effects may distinguish the stress - density relationships found in dynamic experiments from those determined statically. Such effects, although small, have been noted at compaction rates and stresses that are much smaller than those which obtain in our experiments.^{3,6} For stresses greater than a few tenths of a GPa, our experiments suggest that the static stress - density behavior is independent of initial relative density ρ when $\rho < 60\%$. The static compaction of powders having initial relative densities of 47% and 40% should, therefore, be close to that shown in Fig. 5 over the stress range covered by our dynamic experiments. Hence, comparisons between the dynamic and static data obtained here should be representative of the relative compaction rate behavior of these powders.

The dynamic results in Fig. 5 indicate that compaction to a relative density of 65 to 70% occurs readily at stresses below 0.25 GPa, independent of powder type or initial density. This observation is consistent with the rearrangement stage of powder compaction suggested by Cooper and Eaton² and is also apparent in the rapid initial rise of the density with stress seen in the static data. Most of the compaction observed in ceramic powders having initial packing densities below 50% occurs at stresses below 0.3 GPa via this mechanism.² At stresses higher than a few tenths of a GPa, however, densification presumably proceeds via fragmentation and plastic flow. Considerable plastic flow is evident in aluminum nitride specimens compacted with explosives to final densities in excess of 90%,^{10,11} suggesting that such flow is essential to the formation of dense, well-bonded specimens.

For a powder subjected to a rate of loading P , the density is given in general by:

$$\rho = \rho_r (P, \dot{P}^{-1}, t/t_r, t_d/t_r) \quad , \quad (4)$$

where the term \dot{P}^{-1} emphasizes that we expect density at stress P to decrease as the rate of loading increases, and does not necessarily imply anything regarding the actual form of Eq. (4). Time t_r is a relaxation time associated with the densification process, t is the time since P was initially applied, and t_d is the duration or time of application of \dot{P} . For a shock wave, the loading rate \dot{P} is just $\Delta P/\tau$, where τ is the rise time of the stress at the shock front. In porous materials τ is given by D/v_s , where v_s is the speed of the shock and D is the particle size.^{16*}

When different independent mechanisms of consolidation operate concurrently, the right side of Eq. (4) can be written as a sum:

$$\rho = \rho_a (P, \dot{P}^{-1}, t_d/t_a) + \rho_f (P, \dot{P}^{-1}, t_d/t_f) + \rho_p (P, \dot{P}^{-1}, t_d/t_p) \quad , \quad (5)$$

where ρ_a , ρ_f , and ρ_p are the integrated contributions from rearrangement, fragmentation, and plastic flow, respectively. For convenience of notation the dependence on t has been suppressed. The rate dependence of each mechanism will, in general, be different, and their relative rate sensitivities can be assessed crudely by comparing the characteristic relaxation times, t_a , t_f , and t_p , with the loading time, t_d . If the relaxation times are small relative to t_d , the compact is always in mechanical equilibrium and the effect of loading rate is negligible. However,

* Note that through the Hugoniot relationships A-1 and A-2 the density in Eq. (4) can also be written as a function of the linear compaction rate, U_p , rather than P .

when τ_d is small compared with the relaxation times, mechanical equilibrium cannot be maintained and the effect of loading rate becomes significant. Under static conditions τ_d is large and \dot{P} is small so that all mechanisms contribute to the smooth compaction behavior observed as a function of stress. During dynamic compaction the loading time τ_d is just the rise time of the compaction shock τ . Depending upon the mechanism, this may be larger or smaller than the characteristic relaxation times. The particle rearrangement stage, for example, is controlled largely by the inertia of the powder particles, particularly at low initial densities. The time to accelerate an individual particle is roughly the time required to propagate a stress wave through it, D/c_0 , where c_0 is the sound speed of the solid, here about 10 km/s. Hence, inertial contributions to rate effects will be unimportant if $\tau > D/c_0$, or $v_s < 10$ km/s. For this powder in the stress range we have studied, v_s varies from 0.7 to 2 km/s. Particle rearrangement should therefore occur dynamically much as it does statically, consistent with what we observe (Fig. 5). Presumably the other densification mechanisms have longer relaxation times relative to τ , yielding lower dynamic densities at higher stresses. If this is so, the transition from the rearrangement stage should become increasingly abrupt as loading rate increases, as the data in Fig. 5 suggest.

Each point on the Hugoniot corresponds to the maximum rate of compaction which can be achieved in a powder of given initial density at a given stress. Hence, the densities derived from the Hugoniots set a lower bound on the possible values which can be obtained as a function of compaction rate. While this bound is approximately constant in the stress range of 0.25 to 1.8 GPa for the powders we have studied, it increases toward the solid density at higher stresses.^{10,11,32} We have indicated such behavior schematically in Fig. 5. The work of Hoy et al.³² on prepressed compacts with initial

densities of 67% and 76%, however, indicates that densification is not complete at stresses as high as 6 GPa.

VI. Summary and Conclusions

We have presented our measurements of the Hugoniot curves for two unsintered aluminum nitride powders having initial densities of 1.53 g/cm^3 (47%) and 1.30 g/cm^3 (40%), and have compared them with static data. Dynamic stress - density data calculated from the Hugoniot curves show that the relative density behind the first shock is approximately constant at 70% of the solid. Static compaction measurements, in contrast, show a typical, asymptotic increase of density with stress which varies only slightly for initial densities between 1.79 g/cm^3 (55%) and 1.96 g/cm^3 (60%).

From this work we conclude the following:

1. The initial consolidation of the powder proceeds readily to 70% relative density at stresses below about 0.25 GPa independent of initial density. The mechanism in this stress range, particle rearrangement, is insensitive to compaction rate.
2. For stresses between 0.25 GPa and 1.8 GPa, densification is dominated by mechanisms (fragmentation, plastic flow) which are apparently more sensitive to compaction rate. At the linear compaction rates of our experiments, 0.3 to 0.6 km/s, densities are smaller than corresponding static values and are independent of the stress.
3. Secondary shocks contribute to the final density of the compact when densification behind the primary shock is incomplete.

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Appendix. The Rankine-Hugoniot Relationships

Given a material at initial stress P_0 , material speed U_0 , and specific volume V_0 , subjected to a discontinuous shock wave. The stress P , material speed U_p , specific volume V behind the shock, and the speed of the shock relative to the unshocked material, v_s , are related by the following conservation relationships:

$$U_p - U_0 = v_s(1 - V/V_0) \quad (\text{Mass conservation}) \quad . \quad (\text{A-1})$$

$$P - P_0 = v_s(U_p - U_0) (1/V_0) \quad (\text{Momentum conservation}) \quad . \quad (\text{A-2})$$

The specific energy deposited in the material behind the shock front is

$$E - E_0 = (P + P_0) (V_0 - V)/2 \quad . \quad (\text{A-3})$$

Equations A-1 and A-2 may be combined to give

$$v_s^2 = V_0(P - P_0)/(1 - V/V_0) \quad . \quad (\text{A-4})$$

Figure Captions

Fig. 1. Schematic illustration of the sample assembly. The aluminum cover plate was supported on three piezoelectric pins placed symmetrically around its perimeter. Signals from these pins provide a determination of the relative tilt at impact of the projectile and the cover. The thickness of the carbon sensors has been exaggerated for clarity. Their actual thickness at the carbon element is 0.0125 cm. The container was fabricated from 304 stainless steel.

Fig. 2. Scanning electron micrograph of particles of powder I. The powder particles consist of aggregates of 0.1- μ m crystallites. The powder has been dispersed on a backing material to facilitate observation.

Fig. 3. Carbon sensor records for aluminum nitride, shot 140 (Tables II and IV): (a) Front sensor record, showing the stress in the compact behind the initial shock and the much larger stress from the reflection that returns from the back of the assembly. The amplitude of this reflected pulse is somewhat higher than the stress recorded at the rear sensor (b) because of the difference in shock impedance, ρV_s , between the AlN powder and the aluminum plates on either side (Fig. 1) (see Ref. 24).

Fig. 4. Hugoniot curves of aluminum nitride powders having initial densities 1.53 g/cm³ (powder I) (a) and 1.30 g/cm³ (powder II) (b). Unmarked data points are derived from impedance matching. Those marked with a "G" are direct sensor readings which have been corrected for sensor errors if the stress is greater than 2.0 GPa (Ref. 27). The dashed curves were calculated assuming the linear compaction relationship, Eq. (3). The dotted curves were calculated assuming that the powder crushes to solid density immediately upon the application of a stress, regardless of its magnitude. Reshock states on the Hugoniot of solid aluminum 6061 are indicated with an "R." The Hugoniot of solid AlN is included for reference.

Fig. 5. Relative density as a function of the stress. Data points are calculated from the impedance-match Hugoniot data and represent values at stress. The solid curve is calculated from the relationship $\rho_r = 0.685 + 0.017P$ (GPa) and the dotted curves are hypothetical behavior at low and high stresses. The dashed curves are static data obtained for powders having initial relative densities of 55% and 60% (see text).

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Table I. Powder Characteristics

Characteristic	Powder I*	Powder II**
Composition (wt%)	93.0 AlN 4.2 Al ₂ O ₃ [†] 2.0 Free Al 0.06 C	96.0 AlN 3.0 Al ₂ O ₃ [†] 1.0 Free Al
Particle size range (μm)	1-20	1-7
Median particle size (weight basis--μm)	6	2.5
ACP ("solid") density (g/cm ³) ^{††}	3.2	3.2
Packing density (g/cm ³)	1.53	1.30
Surface area (m ² /g)	3.4	3.4

* Materials Research Corporation, Orangeburg, NY.

** Cerac/Pure Inc., Milwaukee, WI, lot number A1120.

† The Al₂O₃ content is obtained by subtraction of the AlN and free Al assays from the total assay of Al in the specimen.

†† Air comparison pycnometer.

Table II. Primary Shock Results for Powder of Initial Density 1.53 g/cm³

Expt.	Type*	Projectile speed (km/s)	Stress (GPa)	Material speed (km/s)	V (cm³/g)	Relative density at stress**
144	Imp.	0.312	0.41	0.184	0.4557	0.67
	G	0.312	0.34	0.289	0.4079	0.75
142	Imp.	0.427	0.65	0.382	0.4293	0.71
	G	0.427	0.61	0.386	0.4073	0.75
143	Imp.	0.518	0.95	0.453	0.4366	0.71
140	Imp.	0.591	1.17	0.512	0.4283	0.71
140	G	0.591	1.07	0.518	0.4028	0.76
145	Imp.	0.652	1.39	0.558	0.4293	0.71
146	Imp.	0.738	1.76	0.620	0.4344	0.70

* Imp. = Impedance matching; G = Direct sensor measurement.

** Relative to the solid density at atmospheric pressure, 3.26 g/cm³.

Table III. Primary Shock Results for Powder of Initial Density 1.30 g/cm³

Expt.	Type*	Projectile speed (km/s)	Stress (GPa)	Material speed (km/s)	γ (cm ³ /g)	Relative density at stress**
155	Imp.	0.323	0.268	0.305	0.4229	0.73
	G	0.323	0.280	0.304	0.4397	0.70
137	Imp.	0.449	0.508	0.414	0.4318	0.71
153	Imp.	0.599	0.994	0.531	0.4851	0.63
	G	0.599	0.94	0.547	0.4512	0.69
152	Imp.	0.749	1.268	0.663	0.4224	0.68

* Imp. = Impedance matching; G = Direct sensor measurement.

** Relative to the solid density at atmospheric pressure, 3.26 g/cm³.

Table IV. Reshock Results for Powder of Initial Density 1.53 g/cm³

Expt.	Stress (GPa)	Material speed (km/s)	γ (cm³/g)	Relative density at stress
144	1.33	0.090	0.4147	0.74
142	1.85	0.124	0.3732	0.82
143	2.3	0.153	0.3698	0.83
140	2.8	0.185	0.3629	0.84
145	3.2	0.210	0.3623	0.85

Table V. Reshock Results for Powder of Initial Density 1.30 g/cm³

Expt.	Stress (GPa)	Material speed (km/s)	V (cm³/g)	Relative density at stress
137	2.0	0.134	0.3791	0.81
153	2.8	0.185	0.4187	0.73
152	3.9	0.254	0.3587	0.86

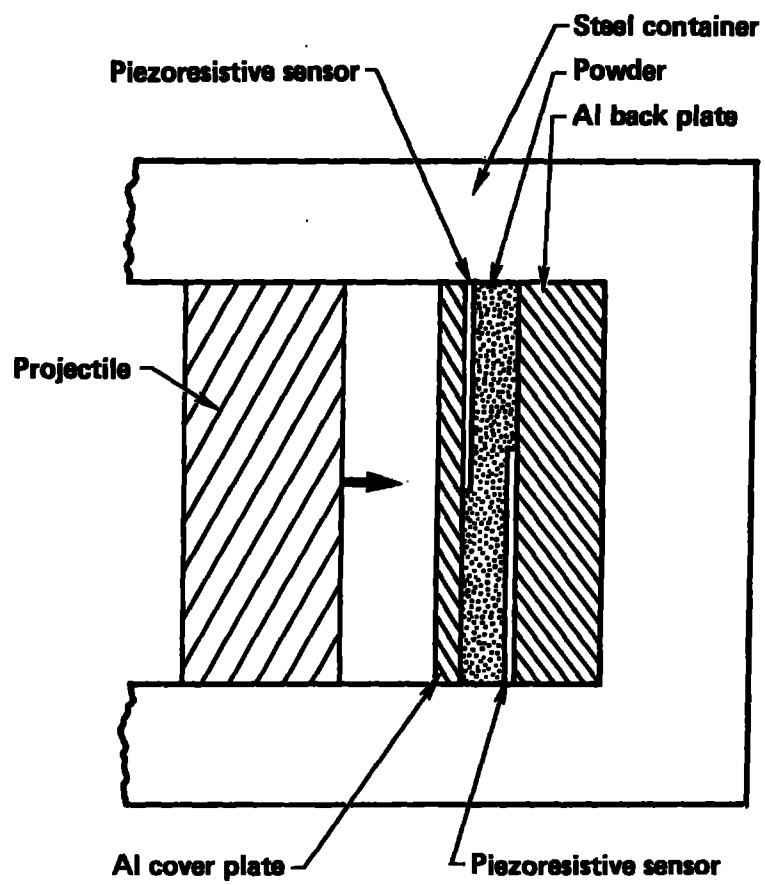


Figure 1



Figure 2

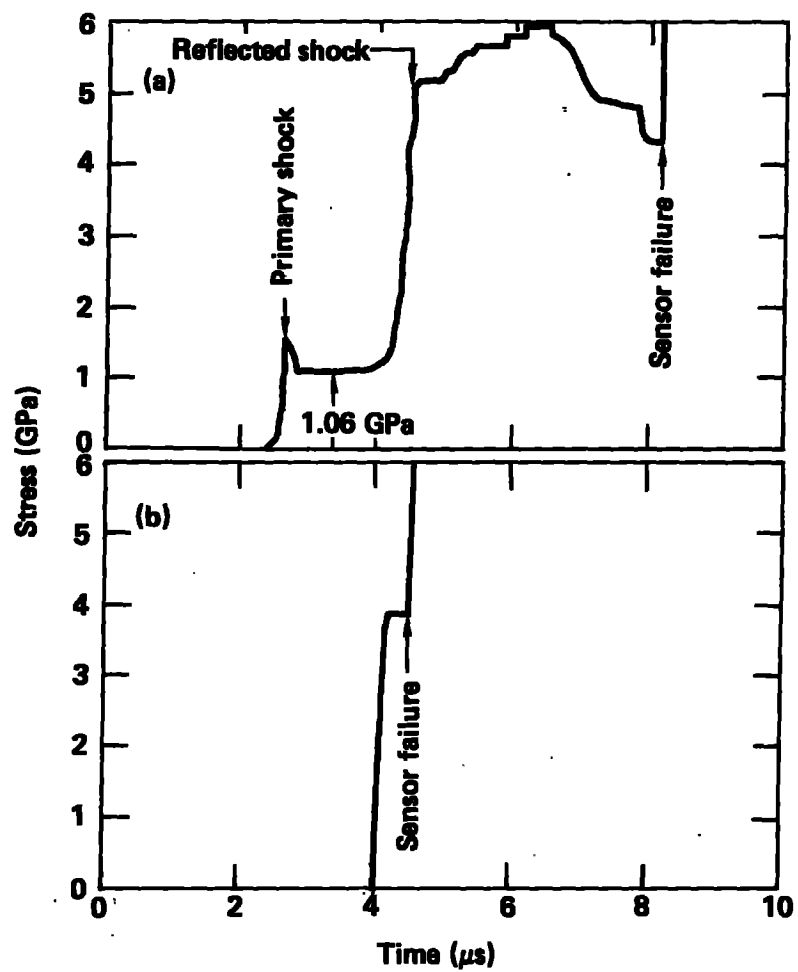


Figure 3

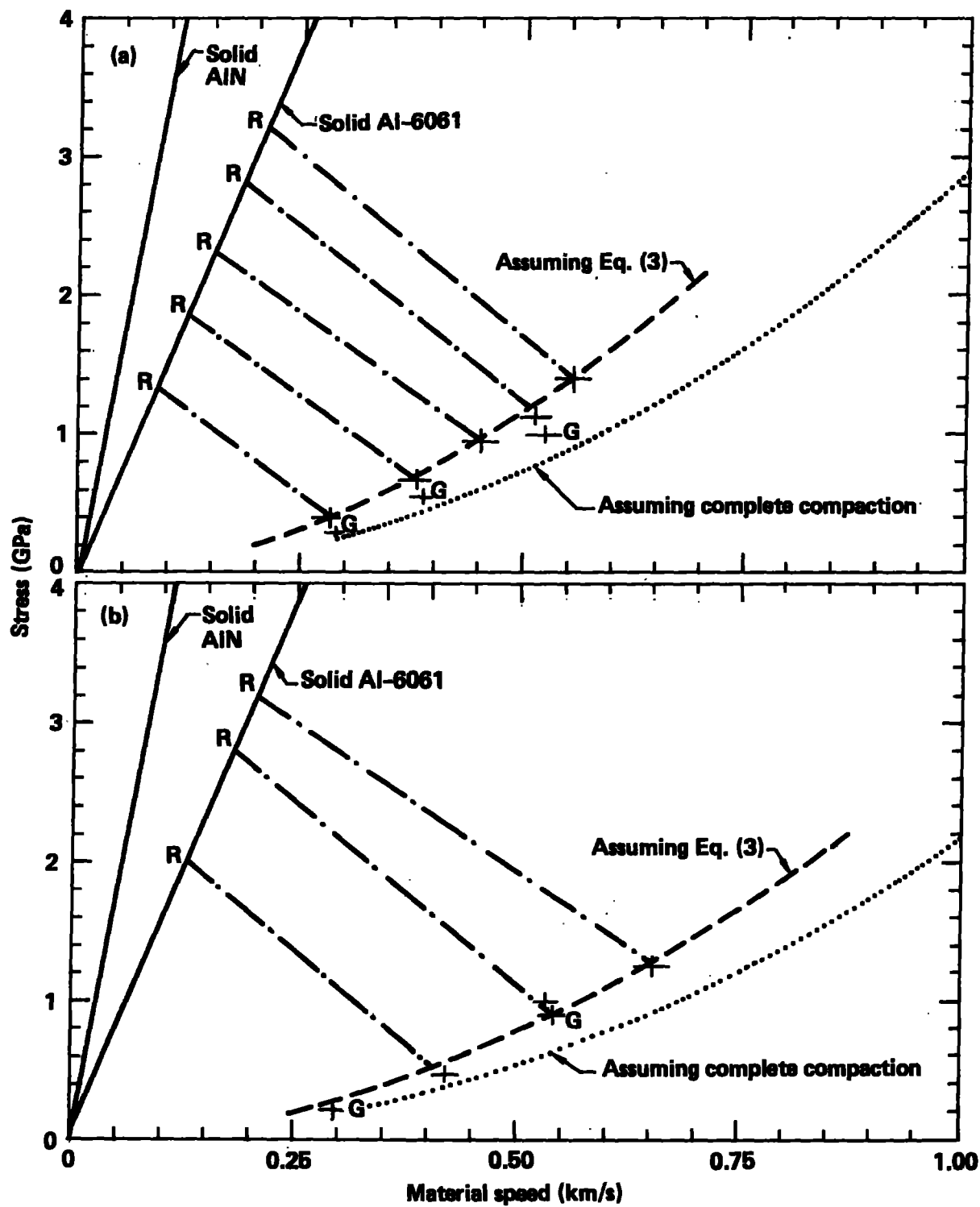


Figure 4

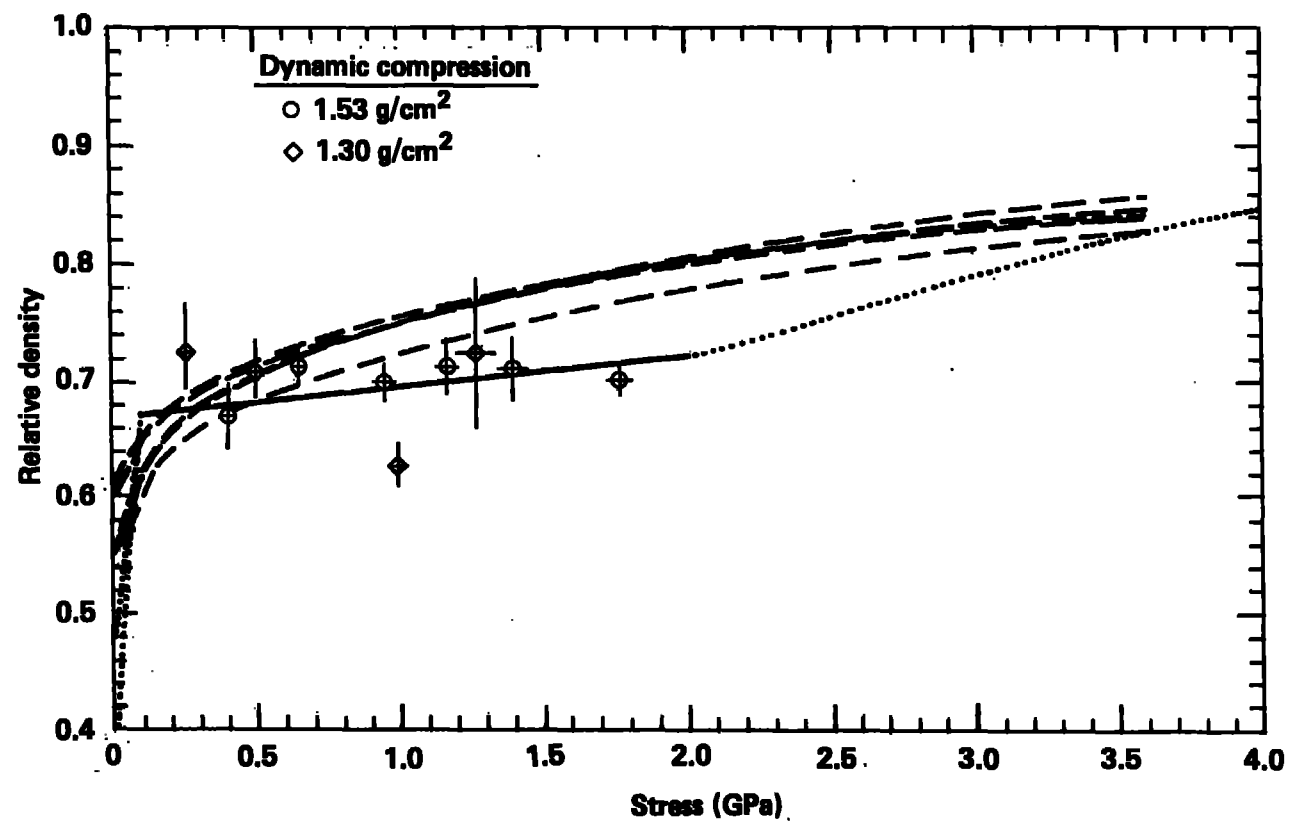


Figure 5